THE NICKEL BULLETIN

A MONTHLY SUMMARY OF CURRENT INFORMATION

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VOLUME 27 NUMBER 5

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ABSTRACTS OF CURRENT PUBLISHED INFORMATION ON NICKEL AND ITS ALLOYS

GENERAL

Nickel Alloys in Needle Thermocouples

W. G. RAUCH: 'Design and Construction of Needle Thermocouples.' *Metal Progress*, 1954, vol. 65, Mar., pp. 71–4.

Certain difficulties arise in using standard two-wire thermocouples to record temperatures of various components of complex heated systems. Such couples require that the two wires be insulated from each other (except at the sensing tip) and that the assembly be enclosed in an impervious metal or ceramic protection tube. The couples are relatively bulky, somewhat fragile and inflexible, and difficult to insert into a confined space. Many of these difficulties are overcome in needle-type thermocouples, which consist of an insulated wire forming one component of the couple, inserted into a tube which represents the second component. The hot junction between rod and tubing is formed by welding. Such couples can be made quite small in diameter by using hypodermic-needle tubing and fine-wire inserts, the insulation being held in place by the tube itself and the tube serving as its own protection. The adaptability and small size of couples of this type make it possible to use them in many locations in which the more conventional forms of thermocouple could not be considered. The material of the tube must be satisfactorily resistant to the atmosphere in which the couple is to be used; the insulation must be flexible, suitable for attachment to fine wire, and resistant to thermal and mechanical damage: the couple combination should have large thermoelectric power, small mass and low heat capacity, and the reproducibility of thermoelectromotive force on repeated heating and cooling must be good. With these requirements in view, the author describes the principles of design and assembly of needle-type thermocouples, and discusses calibration, testing, and operation. He then reports results obtained with various combinations* of tubes and wire, giving calibration curves for the respective couples, compared with curves for Chromel-Alumel couples. Needle-type thermocouples employing the following combinations were studied, the first material mentioned being in each case the tube, the second the wire element: stainless steel/aluminium; stainless steel/copper; Inconel/copper; Kovar/copper; Kovar/Constantan; nickel/Constantan; nickel/copper; nickel/Chromel; copper/Constantan; Inconel/Constantan.

*Chromel: nickel–chromium alloy.
Alumel: nickel alloy containing small amounts of aluminium, silicon and manganese.
Inconel: nickel–chromium alloy containing small percentage of iron.
Kovar: nickel–cobalt–iron alloy.
Constantan: nickel–copper alloy.

Stress in Metal Layers Condensed in Vacuo

See abstract on p. 82.

Production and Processing of Clad Metals

W. L. KEENE: 'Clad Metals and Clad-Metal Processing.' *Steel Processing*, 1953, vol. 39, Dec., pp. 647–53.

Following a brief general discussion of considerations involved in cladding, the author gives a description of some of the principal methods which have been proposed for production of clad metals. The procedures which have proved practicable, and have reached the stage of large-scale development, fall broadly into five classifications, according to the method used, *viz.*, pressing, casting, direct rolling, sintering, deposition. Figures from selected patents covering the respective processes are used to illustrate the descriptions.

The final section of the article contains a condensed review of some of the factors of importance in relation to processing of clad materials, and an indication of some of their major uses.

Porcelain and Ceramic Coatings on Metallic Materials

AMER. SOC. TESTING MATERIALS: 'Symposium on Porcelain Enamels and Ceramic Coatings as Engineering Materials.' *A.S.T.M. Special Technical Publn.* 153, 1954; 122 pp.

Reprints of papers and discussions presented at 56th Annual Meeting of the Society, July, 1953.

The papers covered a consideration of the properties of porcelain enamel and ceramic coatings as related to conditions imposed in various forms of engineering service, industrial processing of such coatings, methods of testing their properties, service durability, etc. Considerable attention is devoted to assessment of the potential value of such coatings in the high-temperature field, when applied to such materials as the nickel- and cobalt-base alloys which are in use for various aircraft power plants. In connexion with torsion testing of enamelled parts, data are presented on the effect of nickel undercoatings of varying thickness.

Use of Complexones in Metallurgical Analysis

E. G. BROWN: 'The Application of the Complexones in Metallurgical Analysis.' *Metallurgia*, 1954, vol. 49, Feb., pp. 101–5 (to be continued).

Since 1945 considerable attention has been given, particularly by chemists in Czechoslovakia, to the applications of the complexones in analytical chemistry, and it is considered that the systematic investigations

carried out by SCHWARZENBACH and his associates have opened a new era in chemical analysis, in which increasing advantage will be taken of the use of ethylenediamine tetraacetic acid (E.D.T.A.) and allied compounds.

In the present survey the author refers to proposals which have been made by various investigators with regard to the application of complexones in specific analyses. The paper is divided into two parts, dealing, respectively, with analysis of ferrous and of non-ferrous materials. The nomenclature proposed by Schwarzenbach is adopted in naming the complexones:

nitrilotriacetic acid, Complexone I;
ethylenediamine tetraacetic acid, Complexone II;
disodium dihydrogen ethylenediamine tetraacetate, Complexone III.

The first instalment of the review deals with the determination of the following elements in ferrous materials: cobalt, chromium, copper, manganese, molybdenum, nickel.

Shell Moulding of Stainless Steel

See abstract on p. 95.

NICKEL

Extraction of Nickel from Missouri Sulphide Ores

H. KENWORTHY and K. K. KERSHNER: 'Metallurgical Investigations of Southeastern Missouri Cobalt-Nickel Resources.' *U.S. Bur. Mines, Rept. of Investigations* 4999, Sept., 1953: 37 pp.

This report gives an account of methods developed for extraction of the nickel and cobalt contents of lead and copper sulphide ores, from concentrates supplied by two lead-mining companies operating in Missouri. Sulphide ore concentrates containing cobalt, nickel, copper, lead and iron have been treated on a laboratory scale to recover more than 90 per cent. of the cobalt, nickel, and copper, and a cobalt-nickel-bearing lead-copper matte has been treated to produce a lead-zinc fume, a copper-sulphide concentrate, and a cobalt-nickel-iron product, with recovery of over 75 per cent. of the cobalt and about 90 per cent. of the nickel and copper. Full details are given of the various experimental methods used, with critical evaluation of procedure likely to prove suitable for large-scale operation.

Electrolytic Precipitation of Nickel from Solutions containing Zinc and Cadmium

J. LAUBER: 'The Electrolytic Precipitability of Nickel from Mixed Solutions (of Nickel) with Cadmium and Zinc.' *Thesis, Eidgenössische Technische Hochschule*, Zürich, 1953; 62 pp.

Electrolytic deposition from solutions of zinc-cadmium, nickel-zinc and nickel-cadmium was investigated, at various temperatures and potential differences. The deposits were analysed polarographically. The paper contains a *bibliography* of 36 references.

Oxidation Characteristics of High-Purity Nickel

E. A. GULBRANSEN and K. F. ANDREW: 'The Kinetics of Oxidation of High-Purity Nickel.' *Jnl. Electrochemical Soc.*, 1954, vol. 101, Mar., pp. 128–40.

The work reported in this paper is part of an extensive study of the mechanism of oxidation of nickel-chromium and nickel-chromium-iron alloys at high temperatures, on which the senior author and his colleagues have been engaged for several years. Reference to relevant earlier papers is given in the *bibliography* attached to the present paper, and the work of other investigators on high-purity metals is summarized.

The purpose of the work now reported was (*a*) to determine the time- and temperature-dependence of the rate of oxidation of high-purity nickel, and how these values are affected by pre-treatment and initial oxide formation, (*b*) to evaluate, from this information, the heat, entropy, and free energy of activation of the rate-determining process, and to compare these values with corresponding data for other metals and alloys, and (*c*) to point out physical and chemical characteristics in the oxide and in the metal which affect the mechanism of reaction.

Using the vacuum microbalance method previously described by GULBRANSEN, reaction of nickel with oxygen was studied over the temperature range 400°–750°C. The results show that nickel oxidizes faster than chromium or 80-20 nickel-chromium alloy: after 30-hour oxidation periods coloured oxide films were formed even at the lowest temperatures within this range.

The data obtained were fitted to the parabolic rate law, and it is shown that the rate law constant decreases with extent of oxidation: the greatest deviations occurred in the range 400°–550°C.

Comparison with results recorded in the literature showed the rate of oxidation of high-purity nickel to be only 1/10–1/1000 of values which have been recorded by other investigators for nickel of a lower degree of purity. The effect of impurities on rate of oxidation is discussed. Using the classic theory of diffusion, entropies and free energies of activation of the rate-controlling process were calculated, and compared with those of other metals. Good agreement observed between theoretical calculations and experimental data leads to the conclusion that diffusion occurs mainly through the lattice of nickel oxide, not at the grain boundaries, at least for the temperature and time conditions in which the experiments were made. The influence of the following preliminary treatments, on oxidation characteristics, was studied: (1) *degassing* (degassed specimens showed increased rates of reaction in the oxidation tests); (2) *electropolishing and annealing* (resulting in strong increase in rate of oxidation at several temperatures), and (3) *pre-oxidation* (a treatment which had a retarding effect on subsequent oxidation). Further work is in progress to elucidate the pre-oxidation effect, which is difficult to reconcile with the interpretation that the oxidation reaction is controlled essentially by lattice diffusion and that calculations of entropy presented

in the paper are correct. It is tentatively postulated that impurities may concentrate in the oxide during pre-oxidation, tending to lower the rate of oxidation at higher temperatures.

Metallurgy for Engineers: Textbook

See abstract on p. 93.

Nickel Powder as Conducting Filler in Welding Primers

See abstract on p. 87.

Stress in Nickel Layers Condensed in Vacuo

H. P. MURBACH and H. WILMAN: 'The Origin of Stress in Metal Layers Condensed from the Vapour in High Vacuum.' *Proc. Physical Soc.*, B, 1953, vol. 66, Nov., pp. 905–10.

The authors report results of measurement of the stress present in deposits of nickel, iron, palladium, gold, copper, silver, antimony, aluminium, bismuth, magnesium, and zinc, condensed from the vapour in high vacuum, on copper-strip substrates. The stress was found to be characteristically of a tensile nature, and to vary in intensity among the various metals. The occurrence, nature, and order of magnitude of the stress are explained as arising from the contraction of the upper regions of the growing deposit as they cool after deposition, which takes place at a temperature generally several hundred degrees higher than the initial temperature of the substrate. According to this interpretation, the contraction will begin to set up a tensile stress in any stratum of the deposit as soon as the temperature of the region concerned falls below the recrystallization temperature of the deposit metal. Estimation of the stress to be expected from this cause falls within the region of the experimentally determined values.

Fabrication of Non-Ferrous Metals in the U.S.A.

See abstract on p. 92.

Use of Complexones in Metallurgical Analysis

See abstract on p. 80.

Effect of Nickel Versenate on Oats

W. M. CROOKE: 'Effect of Nickel Versenate on Oat Plants.' *Nature*, 1954, vol. 173, Feb. 27, pp. 403–4.

Although 2·5 p.p.m. of nickel as nickel sulphate in a nutrient solution will produce marked necrosis in oat plants grown in sand culture, the experiments indicate that the plants do not absorb nickel from the versenate and that they are unable to remove nickel from this complex at the root surface, although it is possible that with iron versenate such mechanism is operative.

Preparation of High-Purity Nickel Chloride

W. S. CLABAUGH, J. W. DONOVAN and R. GILCHRIST: 'Preparation of Nickel Chloride of High Purity.' *Jnl. of Research, Nat. Bur. Standards*, 1954, vol. 52, Feb., pp. 73–4; R.P. 2475.

The method described consists in the repeated precipitation, by hydrogen chloride, of nickel chloride dihydrate, from a mixture of a saturated aqueous solution of nickel chloride with acetone. Details are also given of procedures for determination of cobalt, aluminium, iron, copper and combined phosphorus and silicon, in nickel salts.

Alkali Metal/Nickel Oxides

L. D. DYER, B. S. BORIE, and G. P. SMITH: 'Alkali Metal-Nickel Oxides of the Type $MNiO_2$.' *Jnl. Amer. Chemical Soc.*, 1954, vol. 76, Mar. 20, pp. 1499–503.

The authors describe preparation and properties of two compounds of trivalent nickel which had not previously been isolated: sodium nickelate (III), $NaNiO_2$, and lithium nickelate (III), $LiNiO_2$.

Solid Solutions of Lithium Oxide in Nickel Oxide

P. J. FENSHAM: 'Solid Solutions of Lithium Oxide in Nickel Oxide.' *Jnl. Amer. Chemical Soc.*, 1954, vol. 76, Feb. 20, pp. 969–71.

This paper deals with experiments made in the course of the study of the behaviour of mixed oxides, which have proved to be catalysts of very wide applicability. The effect of gaseous atmosphere and temperature of diffusion, on the solid solution of lithium oxide in nickel oxide, was investigated. The results show that the partial pressure of oxygen present during the solid solution determines its character and extent. Absence of excess oxygen prevents formation of a vacancy-free solid solution. The inter-oxide reaction proceeds to some extent at 600°C., but at that temperature it is probably limited to the surface layers. Even at 1100°C. the reaction within the interior of the oxide particles proceeds slowly.

Nickel in Needle Thermocouples

See abstract on p. 80.

Production and Processing of Clad Metals

See abstract on p. 80.

ELECTRODEPOSITION AND OTHER COATING METHODS

Electrodeposition and Metal Finishing : Fourth International Conference

A list of the papers presented at the Fourth International Conference on Electrodeposition and Metal Finishing is given below, with abstracts of those relating to electrodeposition of nickel and cognate subjects. All the papers, together with discussions, will appear in *Transactions of the Institute of Metal Finishing*, 1954, volume 31.

1. P. LACOMBE: 'The Scientific Basis of Anodic Oxidation Treatments of Aluminium and its Alloys.' 12 pp.
2. J. F. G. HERENGUEL: 'Industrial Methods of Brightening and the Anodic Oxidation of Aluminium and its Alloys.' 11 pp.

3. R. C. SPOONER and D. P. SERAPHIM: 'Phosphoric Acid Anodizing of Aluminium and its Applications to Electroplating.' 18 pp.

Although phosphoric-acid solutions are frequently used in commercial anodizing of aluminium and aluminium alloys for plating, information already published has been concerned almost entirely with specific solutions found suitable for use in individual applications, and no quantitative study of phosphoric-acid treatment over a broad operating range has yet been reported. This paper records results of such a systematic investigation, dealing with the effects (in anodizing of high-purity aluminium) of variations in electrolyte concentration, temperature, time of anodizing, current density and air agitation, on the weight of the coating produced, metal loss, coating ratio, and density of the anodic coating. Comparisons are made with results obtained in sulphuric-acid anodizing.

It was found difficult to electrodeposit, directly onto the phosphoric-acid anodized surface, a satisfactory coating either of copper from a Rochelle copper solution or of nickel from a Watts bath, but deposition of copper from a pyrophosphate solution, as recommended by BENGSTROM (*Trans. Electrochem. Soc.*, 1945, vol. 88, p. 273), gave satisfactory results, and panels thus plated with copper and subsequently nickel + chromium plated showed good adhesion and resistance to attack by salt spray and atmospheric corrosion. It is pointed out that the anodic film formed by the phosphoric-acid treatment, considered as a basis for an electrodeposited coating, has the valuable feature of being itself highly resistant to corrosion, and of effecting partial electrical insulation of the electrodeposit from the substrate. Such insulation should reduce the liability to galvanic action, during service, between the active aluminium and the generally more noble coating. Blistering, which tends to occur with zincate-treated plated aluminium, was virtually absent in the panels plated after phosphoric acid anodizing.

4. R. MONDON and H. E. ZENTLER-GORDON: 'Electrolytic Polishing and Metal Fatigue.' 13 pp.

5. R. PIONTELLI: 'On the Experimental Methods for the Determination of Electrode Polarization.' 6 pp.

6. M. SIMNAD, A. SPILNERS and L. YANG: 'Radiotracer Study of Electrochemical Displacement on Metals.' 6 pp.

7. H. FISCHER and H. F. HEILING: 'Morphology of the Growth of Isolated Crystals in Cathodic Metal Deposits.' 13 pp.

8. U. R. EVANS: 'Corrosion or Protection at Gaps in Metallic Coatings on Steel.' 14 pp.

9. C. WILLIAMS and R. A. F. HAMMOND: 'A Quantitative Adhesion Test for Electrodeposited Chromium.' 22 pp.

10. C. P. BRITTAIN and G. C. SMITH: 'The Influence of Annealing on the Structure and Hardness of Electrodeposited Chromium.' 7 pp.

11. R. L. SAMUEL and N. A. LOCKINGTON: 'The Diffusion of Chromium and Other Elements into Non-Ferrous Metals.' 14 pp.

This paper gives an account of experiments on diffusion of metals into nickel and its alloys from the gas phase: it is particularly concerned with diffusion of chromium and molybdenum.

A specific object of the research was to supplement the field of application of chromium diffusion into ferrous metals. Coatings obtained by this method on steel confer chemical properties similar to those of high-chromium ferritic steels, e.g., the coated articles show good resistance to atmospheric conditions and oxidation at temperatures up to 950°C., and they are resistant to nitric acid. They have, however, limitations in that they are not resistant to hydrochloric or sulphuric acid, which severely restricts their usefulness in chemical-engineering applications. Diffusion of alloying elements into nickel is likely to have practical value in widening the use of diffusion coatings in this field.

The scope of the experimental work reported, and some of the major conclusions drawn with regard to the properties of the treated nickel and nickel alloys, are summarized below:—

Diffusion of Chromium into Nickel: Chromium may be diffused into nickel by several methods which have been found suitable for treatment of iron and steel. For example, vapours of chromous chloride or other chromium halides may be circulated round the metal in a stream of hydrogen, while the temperature is maintained at 1050°C. or above. With certain modifications to the composition of the chromizing powder, the box method described for processing steel can be adapted to treatment of nickel. The articles are packed in contact with a mixture of chromium metal powder, unvitrified kaolin (filler), and ammonium iodide, in a box, which is illustrated in the paper. Ammonium bromide, ammonium chloride, and (with a suitable filler) ammonium fluoride may, alternatively, be used to provide the atmosphere, but ammonium iodide has been found to give the most consistent results.

The diffusion-treated nickel has a smooth, matt, silvery appearance: coating thickness varies in relation to time of treatment (graphically illustrated), and is found to be satisfactorily uniform. The average chromium content of the diffused coating varies from 25 to 43 per cent.: typical figures after various treatments are given. Photomicrographs show three zones present in the diffusion layer, explicable on the basis of the nickel-chromium phase diagram. The chromium-diffused nickel shows good resistance to oxidation at elevated temperatures, even in sulphur-bearing atmospheres: some typical weight variations in air at 950°C. are graphically shown.

Diffusion of Chromium into Nickel Alloys

(1) *Nickel-Copper Alloys:* Nickel-copper alloys respond to methods of chromium diffusion used for nickel: Monel and K Monel can be processed at 1050°–1100°C., but with alloys of high copper content temperatures of treatment must be somewhat lowered. Typical chromium-diffusion coatings on Monel are illustrated.

(2) *Nickel-Chromium Alloys:* Alloys of this group,

e.g., Brightray, Nimonic alloys, Inconel, etc., respond readily to chromium diffusion.

(3) *Nickel-Iron and Other Nickel-base Alloys:* An alloy containing nickel 50, iron 50 per cent. was found to behave, under chromium-diffusion treatment, like nickel, and nickel alloys containing small percentages of silicon, manganese and aluminium gave similar results.

Diffusion of Molybdenum and Molybdenum+Chromium into Nickel: In view of the improved acid-resisting qualities of nickel-molybdenum and nickel-molybdenum-chromium alloys, the production of corresponding diffusion coatings was investigated, by means of (*a*) simultaneous diffusion of the two elements, and (*b*) diffusion of molybdenum into a previously chromium-diffused nickel surface. It was found that, with minor modifications, the methods used for diffusing chromium into nickel can be adapted for diffusion of molybdenum. Details of the nature and properties of the respective coatings are given: they show a good degree of resistance to attack by hydrochloric- and sulphuric-acid solutions. The results (shown graphically in the paper) are broadly in agreement with those reported on the behaviour of alloys of nickel with molybdenum and molybdenum+chromium under similar conditions. In all concentrations of nitric acid corrosion rate of the diffusion-treated specimens was practically nil. The structure of the coatings is illustrated.

Diffusion of Tungsten into Nickel: Tungsten can be diffused into nickel and nickel alloys in the same manner as molybdenum; the coated material is resistant to attack by hydrochloric acid, and the microstructure of the coatings resembles that found on molybdenum-diffused samples. Satisfactory results were also obtained in simultaneous diffusion of molybdenum and tungsten.

The series of experiments thus demonstrates that diffusion of chromium, molybdenum and tungsten, either singly or in combination, into nickel and its alloys, produces coatings which have interesting properties from the point of view of resistance to thermal oxidation and acid attack. The depth of the diffused layers is such as to render the treatment applicable to electrodeposited nickel coatings or to nickel-clad material, as well as to bulk nickel.

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12. W. E. HOARE: 'Recent Advances in Tinplate Manufacturing Processes.' 16 pp.

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13. T. E. SUCH: 'The Prevention of Cracking in Nickel Electrodeposits.' 17 pp.

The paper is introduced by a discussion of the incidence of cracking in electrodeposits (particularly bright coatings) and the inter-relationship of ductility (or brittleness) and internal stress, as influencing susceptibility. Laboratory investigations of cracking have been reported in several papers (to which reference is given), but this work has been done mainly on pure solutions, and very little information has previously been published on the behaviour of the proprietary bright solutions generally used in commercial plating plants. The present paper refers to a study of the subject in the works of Wilmot Breeden, Ltd., where the problem of cracking was approached from two angles: (1) laboratory investigations under closely controlled conditions, and (2) frequent checking of shop plating solutions which were being continuously worked.

The first part of the paper is concerned with equipment and technique developed for determination of internal stress (curvature of thin strip – cathode method) and ductility (tensile tests on nickel-plated steel specimens, with observation of stage at which the plating cracked). An account is then given of tests made, by the methods described, on deposits prepared from several types of plating solution, including proprietary organic and cobalt bright-plating baths, proprietary organic and formate semi-bright baths, and Watts dull-nickel solutions. The results show that a wide range of stress and of ductility may be found in deposits from any one type of bath, without any obvious change in composition or operation conditions. On the basis of the data recorded, suggestions are made for production control of plating solutions by the methods described in the first part of the paper, and experience of the use of such control procedure is demonstrated by a report on the case history of operation, over a three-month period, of an organic bright nickel solution (2,200 gallon-vat) in which a special anti-stress chemical is present.

Some suggestions are made for remedial treatment of production plating vats under routine plating conditions if, as determined by the control tests, it is shown that (*a*) the internal tensile stress has risen, while ductility remains good, (*b*) ductility has fallen to a very low value or the stress/ductility ratio approaches the critical limit above which cracking is likely to occur. (The existence of this critical limit is discussed in the paper.) The observations made by the author have proved that fully bright organic nickel deposits may be almost as ductile as dull deposits, but that ductility is liable to fall off as the solution is worked, and that further investigation is needed to evolve a bright bath which shall be free from this major disadvantage. Such a development calls for closer fundamental knowledge of the interaction of compounds, both inorganic and organic, with the nickel being deposited.

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14. H. LEIDHEISER: 'Studies of the Action of Organic Compounds at the Surface during the Electrodeposition of Nickel.' 16 pp.

This paper reports further aspects of work in progress at the Virginia Institute for Scientific Research, Richmond, Virginia, U.S.A.: see earlier paper abstracted in *Nickel Bulletin*, 1954, vol. 27, No. 1, p. 3. The organic compounds of which the influence was determined are listed in that abstract.

Three types of experiments were made to elucidate the action of the compounds: (1) X-ray diffraction analysis of nickel deposits made from solutions containing the respective addition media, (2) cathode-potential studies, and (3) adsorption studies. The basis solution used was of the Watts type, containing

(g./L.) nickel sulphate 240, nickel chloride 45, boric acid 30.

It was found that the organic compounds used differed in their ability to alter the grain size and the degree and type of preferred orientation. In general, ductile deposits were associated with a (100) preferred orientation and brittle deposits with an orientation other than (100). The ability of the organic compounds to increase the cathodic polarization was dependent on (*a*) the acidic or basic character of the compound, (*b*) the size of the molecule, and (*c*) the number of available pairs of electrons in the molecule. Available pairs of electrons are defined as those which are capable of being shared with other atoms through association or chemical reaction without breaking completely the original bond between the atoms involved. Preliminary measurements were made of the adsorption of the organic compounds from solution on single crystals of copper, by analysing the degree of ellipticity imparted to plane-polarized light upon reflection from the surface. The adsorption measurements were correlated with the cathode-potential data.

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15. R. SALTONSTALL: 'Bright Nickel Plating: A Review of Progress.' 8 pp.

The paper opens with some statistics on the size of the electroplating industry in the U.S.A., with particular reference to its importance in relation to the automotive industry. The requirements laid down in 1934 by ECKLEMAN, as desirable in bright nickel plating baths and the deposits made from them, are then considered, individually, vis-à-vis progress which has been made in the intervening period in realization of these ideals. Among the points thus critically discussed are the following:—

Characteristics of the Electrolyte: stability and sensitivity to contamination; operating range (pH, temperature, concentration, etc.); anode and cathode efficiencies; ability to produce bright deposits over a wide range of current density; throwing power; simplicity of control, correction and purification.

Characteristics of the Deposit: brightness in relation to thickness; adherence; porosity, ductility, protective power, and production of stress-free deposits; coverage with chromium; hydrogen absorption; freedom from nickel hydroxide.

In conclusion, a note is added on current bright nickel-plating practice in the U.S.A., which is still restricted, by supply conditions, from full potential development. It is emphasized that while for mild conditions partial replacement of nickel by copper, use of synthetic organic coatings and other means have been more or less successful in tiding over the emergency, for outdoor exposure in industrial atmospheres there appears to be no satisfactory substitute for heavy nickel plate.

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16. M. B. DIGGIN: 'Nickel Plating from the Sulphamate Solution.' 14 pp.

Early work on sulphamate electrolytes is reviewed, and an account is given of some recent developments. The nature and properties of sulphamic acid are discussed, with particular reference to its application in electrodeposition; references are given to the relevant basic literature. The introduction of sulphamate solutions in nickel plating dates from the work of Barrett in 1950, which directed attention to the suitability of such baths for use in the electrotyping industry. Brief details of his work are given.

The paper is concerned with the study, in the laboratories of Hanson-Van Winkle-Munning Company, of two solutions of the sulphamate type, the compositions of which are given below:—

	Bath No. 1		*Bath No. 2*	
	oz./gal.	*g./L.*	*oz./gal.*	*g./L.*
Nickel sulphamate	40	300	40	300
Nickel chloride ..	4	30	4	30
Boric acid	4	30	4	30
Naphthalene 1, 3, 6-trisulphonic acid	—	—	1·0	7·5

Details of preparation of the solutions are given. Normal operating conditions are:—temperature 25°-70°C. (75°-160°F.); current density 20-140 amp./sq.ft.; pH (electrometric) 3·5-4·2; agitation by air from a low-pressure blower; continuous filtration.

The influence of the respective constituents of the baths is noted, with particular reference to that of naphthalene 1, 3, 6-trisulphonic acid, which exercizes a marked effect on the properties of the nickel deposit, increasing hardness, lowering internal tensile stress and even creating compressive stress, raising tensile strength and increasing electrical resistivity. These effects are demonstrated by experimental data recorded in the paper. The effects of variations in temperature, current density, pH and composition of the bath, on the structure and physical and mechanical properties of the deposits are studied at some length, and the results of the various series of experiments are shown in tables, graphs, and photomicrographs.

The combination and range of properties obtainable in deposits made from the sulphamate bath, particularly the modified (No. 2) type, have found useful application in three fields of electrodeposition: (1) for re-sizing of worn parts, (2) for production of heavy coatings for various purposes, and (3) in electroforming.

In salvaging of worn parts chromium plating deposited to the thickness required, although hard, exhibits high internal tensile stress, which may vitiate service life. Improvement can be effected by using, for at least a major portion of the build-up, nickel deposits from the sulphamate baths, in conditions adjusted to produce high hardness (and accompanying compressive stress). In some cases it is advantageous to replace the hard chromium entirely by this type of hard nickel deposit. In addition to the controllable hardness and favourable stress characteristics, the fact that, by using sulphamate baths, high hardness is obtainable without abnormal brittleness is an important factor in quality printing with long press runs. Favourable reports on such deposits have been made by the printing industry. The possibility of varying the hardness during the period of electrolysis has been found useful in plating certain aircraft parts in which fatigue values are critical and where an initial soft coating followed by a harder outer one is desirable.

Electroforming is a third important application in which the closely controllable physical properties of the deposits can be utilized to great advantage: its first use in this connexion was in the electroforming of stampers for the gramophone-record industry, and recently it has been adopted for electroforming of geometric surface-roughness standards, for radar plumbing, and for electroforming of aviation devices in which definite electrical-resistivity properties are essential.

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17. A. K. GRAHAM: 'Notes on Cleaning Cycles for Plating.' 8 pp.

Among the outstanding conclusions to be drawn from atmospheric-exposure tests carried out under the auspices of the American Society for Testing Materials are: (1) the protective value of a double nickel coating under chromium (see *Proc. Amer. Soc. Testing Materials*, 1953, vol. 53, p. 256)*, and (2) the relation between the preparatory cleaning cycles used and the performance, on weathering, of the electrodeposited coatings (*Proc. Amer. Electroplaters' Soc.*, 1947, vol. 34, p. 74). The present paper records experimental data obtained in establishing the cleaning cycles used in preparation of the panels for the A.S.T.M./A.E.S. exposure tests. Full details are given of cleaning procedures studied in connexion with plating copper or nickel on steel, and plating nickel on a copper strike, or on buffed electrodeposited copper or nickel.

As a criterion of the suitability of the cleaning cycles used, from the point of view of their influence on the quality of the deposit, a 'peel' adhesion test was employed, a method which was considered to be particularly suitable in that adhesion has been found to be sensitive to many cleaning-cycle variations. Full particulars are given of operation of the 'peel' test, also of a grinding-wheel test employed in some cases by the author. The latter was found to be less severe than the 'peel' method.

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18. W. A. WESLEY and B. B. KNAPP: 'The Weathering of Nickel-Chromium Electrodeposits.' 18 pp.

The paper opens with a brief summary of views held in the earlier years of this century with regard to the possibility of ultimate development of methods for production of pore-free electrodeposits. It is pointed out that the doubtful validity of methods used for testing of porosity has thrown some doubt on the properties of the coatings to which they have been applied.

The authors then report various series of tests carried out, in collaboration with the American Society for Testing Materials and the American Electroplaters' Society, to study the nature of porosity and the mechanism of corrosion of electrodeposited coatings.

Preliminary experiments were made with electrolytic nickel foil (0·45–1·1 mil. thick), on which porosity determinations were made by a photographic method, before and after exposure of the foil to (1) the conventional ferricyanide reagent, (2) the regular hot-water test, and (3) atmospheric exposure. The data thus obtained, together with examination of hundreds of cross sections of nickel deposits, at magnifications of 100 to 500, indicated that 'no conclusive evidence existed that nickel deposits 1 mil. thick, prepared with ordinary care, in accord with good laboratory practice, and never subjected to corrosive attack, need contain any pores'. The presentation of these views, with other supporting evidence, led to the initiation, by the American Electroplaters' Society, of a Research Project specifically concerned with the study of the nature of porosity. The International Nickel Company, a collaborator in this project, undertook investigations of the behaviour of nickel and nickel+chromium coatings under atmospheric-exposure conditions. Weathering tests were made in two locations, representative, respectively, of industrial air and of marine conditions contaminated by atmospheres resulting from industrial towns some 30 miles distant. Since it was considered that differences in behaviour of wrought nickel and of electrodeposited foils should be significant in studying the mechanism of corrosion, wrought nickel-clad steel was included, for comparison with nickel foil and electrodeposited coatings. Specimens of wrought commercial nickel strip, in the cold-rolled and the soft-annealed conditions, were used as weight-control standards.

Information compiled from the many specimens exposed is presented in this paper. The data show that in the industrial atmosphere the behaviour of electrolytic nickel and wrought nickel foils was closely similar, both in respect of loss of weight and number of perforations, but that in the marine atmosphere wrought nickel corroded at a higher absolute rate, though it proved more resistant than electrodeposited nickel to penetration by corrosion. In both types of atmosphere nickel-clad steel showed outstandingly good resistance to perforation, but at an early stage of the exposure this material assumed a yellowish stain suggestive of rusting, which is believed to have been due to iron-contamination of the nickel cladding, favouring rusting and uniform corrosion rather than penetration. Tests made to improve the resistance of electrodeposited nickel coatings to penetration by a diffusion anneal proved unsuccessful.

A later series of tests was designed to investigate the behaviour of coatings typical of those used for decorative purposes, i.e., chromium-plated materials. The specimens selected for chromium plating comprised three thicknesses of nickel foil (made by cold-rolling electrodeposited nickel strip), and commercial wrought nickel foil. The structures of the respective materials are illustrated. Examination of these specimens, after periods varying up to 3½ years, demonstrated that the microstructure obtained by working the basis metal, and its purity, are factors conducive to resistance of chromium-plated nickel to penetration by corrosion.

In view of the probability that purity of the nickel was a factor of importance in relation to corrosion-penetration, and in the light of the work by VERNON on the cause of fogging and methods of inhibiting it, some tests were later made on a group of nickel foils electrodeposited (*a*) from an uncontaminated bath, and (*b*) from baths to which various amounts of lead, cobalt, iron, copper, sulphur, or arsenic had been

* *See also paper No. 28.*

singly added. Exposure of the foils, for a relatively short period in sheltered conditions, showed no marked inhibition of corrosion by any of the alloying elements added. Sulphur accelerated weight loss. Perforation of the foils was found to be more sensitive to variations in composition: sulphur and lead had an accelerating effect, and iron a remarkably suppressive effect. A more comprehensive series of tests, using chromium-plated 'contaminated nickel' foils, as well as foils of bare nickel, confirmed that the alloying elements present in the electrodeposited nickel exerted no inhibitive effect on the total-corrosion reaction. Sulphur again increased the rate of corrosion, and the number of perforations: cobalt and arsenic appeared, in this series, to have a mildly accelerating influence on rate of perforation: iron drastically lowered the rate of penetration. The effect of the chromium surface film (10 micro inches) proved helpful during the first few months of exposure; later it caused great acceleration in rate of perforation, while at the same time halving the overall reaction as measured by weight loss. The influence of the alloying elements in the nickel was thus overriden by the effect of the layer of chromium.

In critical discussion of the various tests, the authors conclude that it is not necessary to postulate the existence of pores in electrodeposited nickel. Breakdown of the chromium film may be represented as a time-consuming process, after which localized corrosion of the nickel layer occurs, due to operation of the nickel/chromium galvanic cell. The patterns developed by this surface pitting were found to be extremely varied with respect to number, size, and shape of pits. Residual stresses present in the chromium, and also possibly in the nickel, were an important factor influencing the type of pitting, as were also the microstructure and chemical composition of the nickel. The results of the tests make it clear that mere increase in the thickness of the nickel layer on plated steel above 2 mils. will not guarantee permanent durability of an electroplated finish. Progress in development of improved nickel+chromium coatings should probably be sought along the lines of methods of delaying penetration of the nickel layer, and alteration in the type of pitting. It is suggested, to this end, that study should be made of the possibly beneficial influence of stress-relief heat-treatment after chromium-plating, purity and structure of the nickel, anti-pitting reagents, greater thickness in the final chromium coating, and alloying of the nickel used as final coating or underlay.

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19. J. T. O'REILLY: 'Automotive Organic Finishing.' 15 pp.

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20. J. TOLE and L. HULIN: 'Infra-Red Stoving for Paint Finishes.' 10 pp.

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21. A. J. ELLEMAN and N. D. P. SMITH: 'A New Type of Primer for Resistance Welding.' 12 pp.

This paper reports work done in the laboratories of Imperial Chemical Industries Ltd., Paints Division, comprising a survey of alternatives to conventional electrically-conducting welding primers, which have the inherent disadvantage that the very high proportion of conducting filler needed to give conductivity adequate for welding imposes severe limitations on the composition of the primer. In most of the primers at present on the market the required conductivity is obtained by the use of zinc dust, but the work described in this paper has shown that nickel powder is a much more effective conducting filler than zinc. The nickel particles, by virtue of their mutual magnetic attractions, form chains of low electrical resistance, whereas with non-magnetic metal powders the inter-particle attractions and the probability of inter-particle contacts are much less. Tests on various metal powders, and on nickel powders having various physical characteristics, led to selection of a powder produced from the carbonyl as being the most suitable for the purpose. On the selected material (grade B powder) tests were made to determine the relationships between powder-grinding time, degree of dispersion of the powder, rate of settling and electrical resistance of the films. Welding tests were made on panels coated with the film media thus prepared.

The results of the tests show that it is possible to make, with nickel powder in suitable physical condition, primer coating media which, while containing only moderate proportions of the metal powder, have conductivity adequate for welding. The invention has been covered by patents (*Brit. Pats.* 697,462 and 705,166). The nickel content of the primer appears to have no adverse effect on the protective qualities of the coating, and it is considered that the use of such media shows technical promise. The major difficulty is that the powder, in common with most other metallic fillers, tends to have a high rate of settlement, but it is believed that this problem may be capable of solution, by improvement in the form of nickel dust used and by other modifications of technique.

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22. E. C. J. MARSH: 'Organic Finishes on Electroplated Coatings, with Special Reference to Adhesion.' 16 pp.

The most important reason for using a combination of electroplated coatings and organic finishes is to provide better resistance to corrosion than would be afforded by either finish alone. There are, however, other reasons for which dual coatings are selected, e.g.,

(1) to give improved adhesion to the organic coating, e.g., nickel on brass and phosphor bronze,
(2) to provide electrical insulation, e.g., enamel on electroplated cable supports,
(3) to afford outstanding contrast, as in various types of name plate,
(4) to satisfy electrical requirements, where electrical contact has to be made to the basis metal, as in panels and covers for electrical apparatus,
(5) to utilize raw material which is supplied in the electroplated condition, e.g., zinc-plated steel sheet.

This paper is written from the practical standpoint of the manufacturer who applies such finishes, and the information given is based on procedures developed, in production, to solve difficulties encountered in

fabrication or assembly, or to satisfy service requirements.

The author deals first with application of organic films to *zinc-plated* parts, where a number of pre-treatments have been tried as an aid to adhesion. The solution to this problem has been found in the introduction of an 'etch primer', developed some years ago by the Bakelite Corporation of America. This medium is based on vinyl-butyral resin, and is provided in two separate solutions. One of these contains the film-forming resin and a special form of zinc-chromate inhibitive pigment, zinc tetrahydroxy-chromate, the solvents being of the alcoholic type. The other solution contains phosphoric acid in alcoholic solvents. The two solutions are mixed in specific proportions, and the primer must be used within a limited time after mixing. Suitable application of a correctly selected grade of the etch primer ensures, in the final organic finish, a degree of adhesion infinitely superior to that obtainable by the use of chromate or phosphate treatments.

Application of organic finishes on *cadmium plating* presents even greater difficulties; best results have been obtained by overcoating with a thin electro-deposit of tin prior to application of the organic finish. With *copper plating* light sand-blasting prior to application of the organic finish has effected some improvement in adhesion, but the most satisfactory results are obtained by adoption of the etch-primer technique. In coating *tinplate* with organic finishes, cellulose media pigmented with aluminium are sometimes used as primer coats for cellulose-enamel finishes, and stoved finishes are reasonably good, but in both cases improvement out of proportion to the work involved results from the use of an etch primer, which method has also been satisfactorily adopted to solve the difficult problem of securing adherent organic films on *chromium-plating*.

The application of organic finishes to nickel plating does not present major difficulties; enamels and lacquers adhere satisfactorily. Glyptal-resin lacquer gives an excellent thin film on dull or polished nickel, protecting it against fingermarking, retarding the 'yellowing' of the nickel by normal atmospheric oxidation, and against 'greening' under more severe conditions of humidity, condensation and exposure in industrial atmospheres. The ordinary cellulose enamels, when applied to nickel plating, exhibit their normal shortcomings, i.e., gradual ageing and shrinkage, with associated embrittlement and loss of adhesion, but this can be prevented by the use of an etch primer, which ensures continued adhesion. Stoved coatings give reasonable life, provided that they are selected in relation to the service for which the component is intended. Nickel plating has, indeed, been widely used as a surface preparation for copper-rich non-ferrous metal parts prior to enamelling, but the etch-primer method would now be preferred.

In the radio-communications field, *silver and gold plating* has to be enamelled, particularly the former. Stoving enamels show reasonable adherence to these coatings, but for resistance to severe conditions the etch primer is again recommended. The value of this latter method of preparation is exemplified by the results of corrosion tests on stoved-enamel specimens of cadmium and zinc on steel, and of tin, nickel and nickel+chromium on brass, each type of plating being coated with the organic finish (*a*) after treatment with the etch primer, and (*b*) without such pre-treatment. The results give convincing proof of the value of the pre-treatment, e.g., in the cases of the nickel and nickel+chromium coatings, the enamel on the non-treated specimens blistered in one day in a cyclic hot-humidity test, whereas specimens which had been treated with the etch primer were unaffected after 21 days' exposure.

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23. F. A. LOWENHEIM: 'Tin-Alloy Plating: American Experience.' 12 pp.

The author opens with a short review of factors operating in the U.S.A. which are (1) unfavourable, or (2) favourable, to the development of alloy plating, and gives an account of recent progress and experience in that country in the use of tin-zinc, tin-nickel, and tin-copper coatings. Reference is also made, more briefly, to U.S. experience with tin-lead, tin-cadmium and ternary tin systems in the electroplating field. With regard to tin-nickel alloy plating, American experience is not yet so far advanced as with some other tin-alloy platings, but this form of coating has already aroused much interest and is in operation in one laboratory on a commercial scale. Comments, based on the author's own experience, and on information gleaned from others in the U.S.A. who are experimenting with the process, include the following:

'The principal disadvantage of the plate is its lack of full brightness, necessitating colour buffing for best results. The seriousness of this drawback should not be exaggerated, however, and it varies greatly, depending on the particular application. As to the operational features of the process, our own experience justifies the conclusion that it is eminently practical when conducted with reasonable care and skill. Comparative tests of the corrosion-resistance of the tin-nickel deposit are under way in America, but only isolated results are available. Such observations as have been made are for the most part favourable, but most of these are based on the salt-spray test or some variation of it, and more confidence can be felt when results of outdoor exposure tests, now barely started, begin to come in. On the laboratory scale, barrel plating has been done with no trouble whatever, and the one commercial installation in America at the present time is a barrel of fair size which has been in operation nearly two years and is giving complete satisfaction.'

Particular attention is directed to the fact that many potential users of tin-nickel plating in America have insisted that for maximum usefulness it must be capable of being chromium plated. Contrary to the general experience with deposits of tin or high-tin alloys, the author's laboratory has found that there is little difficulty in depositing chromium over tin-nickel alloy: the only critical factor appears to be the acid dip before deposition of the final chromium. Considerable interest in the tin-nickel alloy coatings arises from the saving in nickel thus secured.

24. A. E. DAVIES: 'Progress in Tin-Nickel Electroplating.' 15 pp.

Since the introduction of bright tin-nickel electroplating in 1951, important advances have been made: major technical problems associated with the operation of the process have been solved and control has been simplified. In 1951 PARKINSON presented data demonstrating that tin and nickel are deposited in roughly equiatomic proportions from solutions coning stannous chloride, nickel chloride, sodium fluoride, and ammonium bifluoride. Subsequent work has shown that deposition of these alloys is possible from several solutions which do not contain chloride, but that fluoride must always be present. Alternative solutions are sulphates, fluoborates, and fluosilicates: detailed information on baths of these types has been embodied in technical and patent publications, to which reference is given.

The present paper reports an investigation of the rôle of the fluoride ion in the chemistry of the tin-nickel electrolytes and in the deposition of tin-nickel alloys. Extensive details are given of the experimental work, and the practical applications of the results are summarized as follows:—

For successful deposition of bright tin-nickel alloy the following conditions must be met:—

(*a*) Ni^{II} concentration should be at least 40 g./L.
(*b*) Sn^{II} concentration should be about 25 g./L.
(*c*) F^- concentration should be sufficiently high to allow for the complexing of all Sn^{2+} and Sn^{4+} ions as SnF_4^{--} and SnF_6^{--} respectively.
(*d*) pH should be about 2·5.
(*e*) Temperature should be between 65° and 70°C.
(*f*) The electrolyte should be continuously filtered.

The major disadvantages of the solution originally proposed by Parkinson were the high hydrofluoric-acid concentration, and the absence of adequate control over the build-up of stannic tin in the electrolyte. Further experiments have shown that hydrochloric acid can be used in place of hydrofluoric acid, without adverse effect on the quality of the deposit: a high concentration of acid is unnecessary. Two modified tin-nickel solutions now suggested are shown below:—

Solution Compositions

	A g./L.	*B* g./L.
$NiCl_6.6H_2O$	250	250
$SnCl_2.2H_2O$	50	50
NH_4F	50	33
NaF	—	20
HCl (32% w/w)	8	8

Recommendations are made on maintenance of the two solutions, which are now undergoing tests in a 40-litre pilot plant.

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25. G. E. GARDAM and N. E. TIDSWELL: 'The Electrodeposition of Gold and Other Alloys by a New Method.' 8 pp.

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26. R. A. SCHAEFER, J. F. CERNESS and H. A. THOMAS: 'The Resistance of Some Cast and Plated Sleeve-Bearing Materials to Cavitation-Erosion.' 10 pp.

27. W. A. BELL: 'A Laboratory Technique for the Electrodeposition of Manganese on Other Metals.' 10 pp.

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28. W. L. PINNER: 'Unsolved Problems in the Metal Finishing Industry.' 6 pp.

The author opens by emphasizing the tremendous strides which have been made during the past thirty years in the electroplating industry. Improved nickel-plating baths have been introduced; chromium plating has been commercially adopted; much new knowledge has been gained on cleaning procedures; many analytical control procedures have developed, and the relation of plate thickness to product quality has been more closely evaluated. As in other industries, progress has also resulted in an increased awareness of problems still standing in the way of consistent large-scale output of products of the highest quality, at the lowest possible cost.

Improvement of product must be based on a full understanding of the chemical, electrochemical and physical factors which affect deterioration of the finished product, and full evaluation of the product demands reliable test criteria. Service exposure is at present the only reliable approach, and in this connexion the author refers to information which has been obtained as a result of the atmospheric-exposure tests in progress under the aegis of the American Society for Testing Materials and the American Electroplaters' Society. Particular attention is directed to two sets of panels which have given outstandingly good resistance to corrosion: (1) those plated with nickel to an average thickness of 0·0005 in., buffed, and subsequently plated with an additional nickel coating of the same thickness, which was likewise buffed and finally chromium plated; (2) treated as set 1, except that the first layer of nickel was annealed after buffing, prior to the further treatment. These two sets of panels are showing remarkable protection of the base metal, in comparison with all other combinations of plate of equal thickness. The present writer urges the importance of a better understanding of the effect of treatment of the basis-metal or undercoating, on the properties of electrodeposited coatings, and of the mechanism by which electrodeposited films deteriorate.

A further urgent need of the electroplating industry is for a valid accelerated corrosion test, to replace the salt-spray test, which has been discredited on the basis of research carried out by the A.S.T.M./A.E.S. Committee.

The influence of the basis metal, on quality and serviceability of electrodeposits, is a further subject which calls for more extensive study. There appears to be need for development of polishing methods which will not result in the creation of fragmented metal, and the author considers that, in the meantime, 'if all operations involved in the fabrication of metal parts be regulated so as to require a minimum of abrasive metal polishing, with as fine a grain as possible, this procedure is likely to result in better quality, which could also probably be achieved at lower manufacturing costs.'

Another unsolved problem to which attention is drawn is the development of organic-type bright-nickel plating baths which will produce deposits showing much higher ductility than is at present consistently attainable from bright plating solutions.

Finally the author suggests that too little is known about the composition and working of cyanide-copper plating solutions, particularly with regard to the chemical linkage of the copper, sodium, and cyanide ions, and the retention of smoothness in any considerable thickness of plate deposited from this type of bath.

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29. S. WERNICK: 'Universal Metal Finishing Problems: A Plea for International Co-Operation.' 5 pp.

The writer points out that although problems which confront the metal-finishing industry of one country are in many cases closely similar to those which are giving trouble in other countries, all too frequently the relevant work carried out (and even published) in one region is but little known in other lands. He believes that a considerable service to all countries would be performed by the creation of 'an International Bureau charged with the task of collecting and collating all publications on metal finishing, irrespective of the source of origin, with the object of cataloguing, abstracting and summarizing the garnered information for subsequent distribution throughout the world'. The main object of the paper is a plea for international action in dealing with serious problems which affect metal finishers throughout the world. Appreciation is expressed of the extensive researches undertaken by the American Electroplaters' Society, but it is urged that the co-operative work should be widened, with European countries making a greater contribution. As typical of problems confronting the industry, the author refers to:—

(1) *Elimination of Roughness in Nickel Plating:* The importance of this subject is discussed in relation to the serviceability of nickel deposits and the economics of production.

(2) *Development of an Efficient Chromium Electrolyte*, which should be characterized by higher cathode efficiency, improved throwing power, lessened toxicity, and the capability of producing low-stress deposits.

(3) *Development of a Reliable Workshop Method for Determining the Thickness of Nickel Deposits.* Although the B.N.F. Jet Test met requirements for matt nickel deposits, the position has been radically changed by the advent of bright nickel plating, for which thickness measurement involves corrections varying according to the nature of the bath from which the coating has been deposited.

(4) *Disposal of Effluents.* Simple and economical methods for disposal of noxious effluents are already under intensive study by the American Electroplaters' Society, but the subject is of such universal importance as to call for co-operative effort in various countries.

To the above problems may be added those enumerated in the paper by PINNER: see abstract on p. 89 of this *Bulletin*.

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30. H. SILMAN: 'Glass-Fibre Reinforced Plastics: A New Material of Construction for the Finishing Industry.' 17 pp.

31. C. L. FAUST and W. H. SAFRANEK: 'The Electrodeposition of Porous Metal.' 10 pp.

This paper gives an account of methods developed for production of coherent porous electrodeposits of copper, nickel and zinc, using colloidal graphite as the pore-inducing agent. In deposition of copper, gelatin was also added to the bath, to improve the dispersion of the colloid. It is believed that the techniques developed can also be applied in production of porous coatings of iron, chromium, tin, and lead.

Using the methods described, it was found possible to deposit sound, smooth and coherent deposits up to $\frac{1}{8}$ in. thick, having porosity as high as 58 per cent. Adherent coatings were applied to steel, and porous discs and tubes were electroformed, starting with steel mandrels coated with graphite as a parting medium. Typical products, of varying porosity and pore size, are illustrated. Such electrodeposits are permeable to both gases and liquids: typical permeability data are quoted, physical characteristics of the electrodeposits are indicated, and their structure is illustrated.

Full details are given of technique for deposition of porous layers of the three metals studied: some particulars of the methods used in production of the porous nickel are given below:—

	Composition of Bath g./L.
Nickel sulphate ($NiSO_4.6H_2O$)	330
Nickel chloride ($NiCl_2.6H_2O$)	45
Boric acid (H_3BO_3)	40
Aquadag (colloidal graphite)	60

The surface tension of the bath was maintained at about 40 dynes/cm. by adding, when required, a solution of purified sodium lauryl sulphate. In the absence of a wetting agent the deposits were pitted.

Increase in concentration of Aquadag over the range 1–50 g./L. resulted in gradual rise in porosity of the electrodeposit: further additions, 50–150 g./L., had the reverse effect. The influence of pH, temperature and cathode current density, on porosity, is graphically illustrated. On the basis of the observations made, the following conditions were selected for electroforming porous nickel tubes:—pH 4·5–5·5, temperature 60°C., current density 100–135 amp./sq. ft. Deposits produced in these conditions were smooth and microporous, and contained 36–58 per cent. of voids, depending on the current density employed.

In operation of both the copper- and nickel-plating baths porosity of the deposits decreased with time, an effect associated with coagulation of the graphite particles. Several other colloidal materials were tested as alternatives to Aquadag, but none gave deposits superior to those obtained with that form of pore-inducing addition.

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32. P. BAEYENS: 'Electroplating with Modulated Current.' 24 pp.

Electroplating is usually associated with direct-current electrolysis, although pure direct current is rarely employed, because some form of ripple is almost always present. In some instances, however, deliberate use is made of other forms of current, e.g.,

alternating current, alternating superimposed on direct current, and periodic-reverse (P.R.) current. The author of this paper makes a detailed review of the present state of development and knowledge on the use of all variations of 'non-direct' current, using, for convenience, the term 'modulated' to describe any current which undergoes some change of value, either absolute or of polarity, which is repeated periodically and of which many cycles occur in one electroplating operation.

After discussion of the variables which must be taken into consideration in use of 'modulated' currents, and installations and technique required for their application, a review is made of the historical development of the principle. The final section of the review contains a summary of information relating to the use of 'modulated' currents in electrodeposition of individual metals: sodium, aluminium, chromium, iron, cobalt, nickel, copper, zinc, arsenic, molybdenum, silver, cadmium, gold, tin, and various alloys.

The review is supported by *110 references to original literature.*

Industrial Electroplating: Handbook

E. A. OLLARD and E. B. SMITH: 'Handbook of Industrial Electroplating.' 2nd Edn.
Published, for Metal Industry, by Iliffe and Sons, Ltd., London; 362 pp. Price 30/–.

The first edition of the Handbook, published in 1947 (see *Nickel Bulletin*, 1947, vol. 20, No. 10, p. 143), was specifically designed to be a practical work of reference for the operator in the plating industry. The authors did not attempt to deal with the theory of electrodeposition, but confined their attention to the practical aspects of installing and maintaining plant for electroplating operations, and the preparation, maintenance, and use of solutions.

The new edition has been considerably amplified, to cover, within the limits indicated above, developments which have taken place during the past seven years. *Inter alia*, it embodies new sections dealing with water and drainage, purification of solutions, safety precautions and ventilation in plating shops, and problems of costing. Formulæ for new or modified solutions are also included and some additional information on testing of electrodeposits is provided. The sections containing a glossary of terms used in the plating industry, and miscellaneous information of a general reference character for electrochemists have been enlarged.

Cleaning of Metals Prior to Electroplating

H. B. LINFORD and D. O. FEDER: 'Cleaning and Preparation of Metals for Electroplating. VIII. Effect of Oxide Films. A Survey of the Literature.' *Plating*, 1954, vol. 41, Mar., pp. 279–86; Apr., pp. 397–401. (Report issued under the aegis of A.E.S. Research Project 12.)

The literature survey summarizes previous work on (*a*) the nature of the problem of cleanness of surface of basis metals on which electrodeposits are made; (*b*) the growth of oxide films, and their characteristics; (*c*) the mechanism of metal deposition, crystal formation, and adhesion. In the light of known facts on these aspects, interfacial phenomena occurring during plating onto an oxide-soiled metal surface are interpreted. The effects of these processes, and of the oxide films on the plated end-product, are evaluated. The review is based on a *bibliography* of 115 references to published literature.

Electroplating of Powder-Metal Parts

R. RUSHBROOK: 'Electroplating of Powder Metal Compacts.' *Electroplating*, 1954, vol. 7, Mar., pp. 92–6.

The porosity (small or large in degree) which is inherent in parts produced by pressing and sintering of metal powders necessitates certain modifications in plating procedure. The author of this article discusses five techniques which have been developed for plating powder compacts, giving details of some typical treatment cycles:—

A. *Plating by conventional methods*, with certain minor modifications of procedure, e.g., for objects of which the porosity is low and the cleaning and plating solutions to be used are not dangerously corrosive to either the basis material or the electrodeposit.

B. *Preventing ingress of liquid to the pores by—*
(1) closing of the pores by mechanical methods,
(2) impregnation of the pores with solids,
(3) impregnation of the pores with water-repellent compounds,
and subsequent plating by conventional methods.

C. *Plating by conventional methods, followed by heat-treatment, to drive off solutions entrapped in the pores.*

The review is supported by references to the relevant technical and patent literature.

Electroless Nickel Barrel Plating

H. J. WEST: 'Electroless Nickel Barrel Plating.' *Metal Finishing*, 1954, vol. 52, Mar., p. 64.

The writer gives full details of the application of the electroless process to barrel plating, for which it has not yet come into general practice. Either high-temperature polystyrene or Lucite was found suitable for the barrels, and a porcelain-lined steel tank gave good results. The size of tank proposed (17 in. deep by 24 in. wide by 45 in. long) with drain and, if possible, steam-heated, accommodates two 6 in. by 12 in. portable plating barrels, which are supported by a plain steel rod.

The formula recommended for the barrel-plating bath is as under:—

800 fl. oz. of 5 lb./gal. solution of nickel chloride.
100 oz. sodium citrate.
100 oz. sodium hypophosphite.

The solution is prepared by adding the chemicals, singly, to water at a temperature of 175°F. (80°C.): complete dissolving must be ensured before plating is begun. The operating temperature for barrel plating is 185°F. (85°C.) and the time of treatment is 30 minutes, which gives a coating thickness of 0·00015 in. pH of the solution should be maintained in the region

of 4, by addition of 2 oz./gal. of a 20 per cent. solution of caustic soda, once during the plating operation, at the rate of about 30 drops per minute.

Thorough cleaning of the parts before plating, and correct drying immediately after plating are essential to success. The writer recommends rinsing in cold water, followed by rinsing in denatured alcohol, centrifuging, dipping in oil, and re-centrifuging.

In order to ensure brightness in the plated parts it has been found necessary to add sodium hypophosphite after four hours' use of the bath; for the solution mentioned above the correct addition is 24 oz. The solution should be discarded after each day's work.

Polarographic Analysis of Nickel-Plating Solutions

J. V. PETROCELLI and G. TATOIAN: 'The Polarographic Analysis of Nickel-Plating Solutions.' *Plating*, 1954, vol. 41, Mar., pp. 262–8.

The work described in this report is part of an extensive investigation carried out in the laboratories of The Patent Button Company, Waterbury, Conn., U.S.A., with the aim of developing polarographic methods for control of some of the more common plating solutions, and of designing a simplified polarographic instrument readily usable by laboratory staff. Following a discussion of the general principles of polarographic analysis, the authors describe (with illustrations) the apparatus developed, and give details of results obtained in the application of the polarographic technique to determination of the principle constituents of a Watts'-type bath and a plain cold nickel-plating solution containing ammonium chloride. Standard curves and detailed procedures are given for estimation of nickel sulphate, nickel chloride, and boric acid. The influence of pH and the effect of brighteners are also discussed.

Cadmium Plating of Stainless Steel

'Cadmium Plating of Stainless Steel.' *Electroplating*, 1954, vol. 7. Mar., p. 99.

In many cases bolts, screws, pins, bushes, etc., made from ferrous materials, which are to be used in contact with light-alloy components in airframe assemblies, are now cadmium-plated, to reduce the risk of contact corrosion of the light metal. In this connexion it is frequently necessary to plate stainless steel.

Due to the passive film on such steels, special plating techniques are essential, and the Ministry of Supply has recently issued R. D. Mat.(A) Memorandum No. 3, recommending the following procedure:—

(1) Solvent degreasing.

(2) Alkaline cleaning, followed by water rinsing.

(3) Pickling in warm dilute sulphuric acid (approx. 10 per cent. by vol.), *or*, particularly if the specified ultimate tensile strength of the material to be plated is above 65 tons per sq. in., anodic pickling in sulphuric acid.

(4) Water rinsing.

(5) Nickel Strike treatment, e.g., the work is treated as cathode at 150–200 amp./sq. ft. for 5–10 minutes in an aqueous solution containing

Nickel sulphate ($NiSO_4.6H_2O$) 225 g./L.
Sulphuric acid (H_2SO_4) 50 g./L.
(i.e., 27 ml./L. conc. acid S.G. 1·84)

The temperature of the electrolyte is maintained at 35°–40°C. (95°–104°F.). Insoluble anodes, e.g., lead, are used.

(6) The part is then given a quick water rinse and is transferred to the cadmium-plating vat and plated immediately in the normal manner. (See D.T.D. 904.)

Porcelain and Ceramic Coatings on Metallic Materials

See abstract on p. 80.

NON-FERROUS ALLOYS

Fabrication of Non-Ferrous Metals in the U.S.A.

ORGANIZATION FOR EUROPEAN ECONOMIC CO-OPERATION: 'Non-Ferrous Heavy-Metal Fabrication in the U.S.A.' *Report prepared by Technical Assistance Mission No. 79*; published Mar., 1954; 253 pp.

The report has been prepared by a Mission of twenty-two specialists, from nine European countries, which visited the U.S.A. in October-November 1951, to study techniques used in the fabricating of sheet, strip, rod, sections, tubes and wire of heavy non-ferrous metals and alloys.

Introductory chapters on conclusions drawn from the tour, and on significant features of the American non-ferrous metals industry in general are followed by reports on individual branches:—refining, melting, and casting of copper; hot-rolling of rods from high-conductivity copper wirebars; melting and casting of copper alloys; manufacture of copper-alloy strip (including cupro-nickel and nickel silvers); manufacture of copper and copper-alloy tube shells, and copper-alloy rods, sections, and wire (including nickel-containing copper-base alloys); production of copper and copper-alloy tubes (including iron-containing cupro-nickel); melting and fabrication of nickel and nickel alloys; fabrication of zinc and lead. A list is given of the firms visited, on whose practice the report is based.

With reference to the fabrication of nickel and nickel alloys, it is pointed out that conditions at the time of the visit were abnormal, due to control and restrictions which had been operating during the war and post-war periods. The information given therefore relates to a narrower group of materials than would usually be in production. Procedure at various plants is described with regard to melting and casting of nickel and some of its alloys, surface preparation, forging, hot-rolling, sheet- and strip-rolling, extrusion, cold-drawing of tubes, bars, and shapes, wire-drawing.

At the end of each of the main chapters of the report there is some discussion of the observations made, with suggestions on certain features of American practice which, in the opinion of the members of the Mission, could with advantage be adopted in European metal-working plants.

Metallurgy for Engineers: Textbook

See abstract on p. 93.

Thermoelectric Power and Resistivity of Nickel-Silicon Alloys

C. A. DOMENICALI and F. A. OTTER: 'Thermoelectric Power and Resistivity of Dilute Cu-Si, Ni-Si, and Fe-Si Alloys.' *Bull. Amer. Physical Soc.*, 1954, vol. 29, Mar. 18, pp. 23-4. (Abstract of paper to American Physical Society, March, 1954.)

'Thermoelectric power and resistivity of the dilute alloys copper-silicon, nickel-silicon, and iron-silicon were measured over the range −195° to +500°C. (Results for the copper-silicon series were reported in *Physical Review*, 1953, vol. 91, p. 220). The nickel-silicon alloys show a knick in the thermopower vs. temperature curve at the Curie temperature of each alloy. The iron-silicon alloys contained silicon up to 2 at.-per cent., and the thermopowers of the iron-silicon alloys against copper are all positive from −195°C. up to a temperature which increases from +15°C. for an alloy containing 2 at.-per cent. silicon, to 250°C. for pure iron. The resistivity of the nickel-silicon and iron-silicon alloys increases faster than linearly with temperature: the change with increasing silicon content is much greater in iron-silicon than in nickel-silicon alloys, but only slightly greater in nickel-silicon than in copper-silicon alloys. Electron scattering in the alloys is being studied, on the basis of their thermoelectric and resistance properties.'

Gold-Nickel Alloys

B. L. AVERBACH, P. A. FLINN and M. COHEN: 'Solid Solution Formation in the Gold-Nickel System.' *Acta Metallurgica*, 1954, vol. 2, Jan., pp. 92-100.

The local atomic configurations measured from diffuse X-ray scattering in the gold-nickel system cannot be reconciled with the observed heats and entropies of mixing on the basis of a statistical treatment of chemical bonding energies. The heats of mixing are positive (heat absorbed), and yet there is a preference for *unlike* neighbours in solutions above the solubility gap. It is shown that these positive heats of mixing may be explained in terms of the elastic strain energy required to form solutions from atoms of different size. The sizes of the ions in gold-nickel solutions, also measured from the diffuse X-ray measurements, are used to calculate the strain energies: reasonable agreement with the observed heats of mixing is obtained.

Nickel-Thorium Alloys

J. V. FLORIO and R. E. RUNDLE: 'Compounds of Thorium with Transition Metals of the First Period. II. The Thorium-Iron, Thorium-Cobalt, and Thorium-Nickel Systems.' *U.S. Atomic Energy Commission Publn.* ISC-273, 1952; 54 pp.
Metallurgical Abstracts, 1954, vol. 21, Feb., p. 526.

A detailed crystallographic study was made of the intermetallic compounds occurring at room temperature in the systems named. The results are summarized in *Metallurgical Abstracts*, q.v.

Nickel Alloys in Needle Thermocouples

See abstract on p. 80.

NICKEL-IRON ALLOYS

Magnetostriction in Nickel Ferrites

D. F. BLEIL and A. R. BUTZ: 'Magnetostriction in the Nickel Ferrite-Aluminates and Nickel Ferrite-Gallates.' *Bull. Amer. Physical Soc.*, 1954, vol. 29, Mar. 18, p. 42. (Abstract of paper to American Physical Society, March, 1954.)

Magnetostriction determinations of the systems nickel ferrite-aluminate ($NiOFe_2$-$_t$$Al_t$$O_3$) and nickel ferrite-gallate ($NiOFe_2$-$_t$$Ga_t$$O_3$) have been made, using a system of compensated strain gauges described by MAXWELL (*Physical Rev.*, 1947, vol. 72, p. 529). The results are briefly summarized in this abstract.

Diffusion of Iron into Nickel at Elevated Temperatures

M. B. NEIMAN *et al.*: 'The Diffusion of Iron in Nickel.' *Doklady Akad. Nauk S.S.S.R.*, 1953, vol. 91, pp. 265-7; *Jnl. Iron and Steel Inst.*, 1954, vol. 176, Feb., pp. 235-6.

'The diffusion of iron in nickel in the temperature range 950°-1130°C. was studied, using radioactive Fe_{59}. Thin nickel foil was plated on one side with iron containing radioactive Fe_{59}, and was heated, in a silica vessel, *in vacuo*, to a given temperature. The rate of diffusion was followed by measurements of the radioactivity on both sides of the foil. The temperature dependence of the diffusion coefficient of iron in nickel, and the activation energy, were determined. The velocity of the diffusion of iron into nickel is considerably higher than that of nickel into iron.'

Nickel Alloys in Needle Thermocouples

See abstract on p. 80.

CONSTRUCTIONAL STEELS

Metallurgy for Engineers: Textbook

B. STOUGHTON, A. BUTTS and A. M. BOUNDS: 'Engineering Metallurgy: a Textbook for Users of Metals.' *Published by McGraw Hill Publishing Co., Ltd.*, London, 4th edn., 1953; 479 pp. Price £2 13s. 6d.

The purpose of the first edition of this well-known textbook, issued in 1926, was to make available information adapted to the needs of students in engineering branches representing users of metals. In the subsequent editions this primary aim has been preserved, and although reference is made to many of the advances in process metallurgy which have taken place in recent years, material on these aspects is abridged in the interests of fuller presentation of major developments in physical metallurgy, the properties of metallic materials, and their utilization. The essential purpose throughout is to present to the student theory and information based on practical experience, which together will provide him with the background for sound application of metals to engineering requirements.

Machining of Nickel-Alloy Steel Crankshafts for Oil Engines

'Machining Crankshafts for Albion Oil Engines.' *Machinery* (*Lond.*), 1954, vol. 84, Feb. 26, pp. 419–27.

The article describes equipment and methods used, by Albion Motors, Ltd., Scotstoun, Glasgow, in production of crankshafts for goods and passenger vehicles, in which this firm specializes, in association with Leyland Motors, Ltd. Particular attention is given, in the description, to methods employed in machining of crankshafts for the 4-cylinder Diesel-type engine EN 286, which is supplied in forms suitable for incorporation in vehicle, industrial and marine engines.

In order to ensure properties adequate for the heavy service in which such engines are employed, the crankshafts are made from forgings of En 24U steel (2½ per cent. nickel–chromium–molybdenum steel of medium carbon content, heat-treated to the 60-ton tensile condition).

Specifications for High-Tensile Nickel-Alloy Steels for Aircraft

BRIT. STANDARDS INSTN.

(1) '55-ton Low Nickel-Chromium-Molybdenum Steel (Limiting ruling section 4 in.).
Specn. S. 118; Feb., 1954,
covering steel of the following composition:—

Carbon 0·35–0·45, silicon 0·10–0·35, manganese 0·4–0·8, nickel 1·2–1·6, chromium 0·9–1·4, molybdenum 0·10–0·20, sulphur 0·045 max., phosphorus 0·045 max., per cent.

Hardening and tempering treatment and mechanical properties are specified: the latter include ultimate tensile stress 55–65 tons per sq. in.

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(2) '65-ton, 1½ per cent. Nickel-Chromium-Molybdenum Steel (Limiting ruling section 2½ in.).
Specn. S. 119; Feb., 1954,
covering steel of the following composition:—

Carbon 0·35–0·45, silicon 0·10–0·35, manganese 0·45–0·7, nickel 1·3–1·8, chromium 0·9–1·4, molybdenum 0·20–0·35, sulphur 0·045, phosphorus 0·045, per cent.

Hardening and tempering treatment, and mechanical properties are specified: the latter include ultimate tensile stress 65–75 tons per sq. in.

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(3) '100-ton, 2½ per cent. Nickel-Chromium-Molybdenum Steel (Oil Hardening) (Limiting ruling section 2½ in.).
Specn. S. 120; Feb., 1954,
covering steel of the following composition:—

Carbon 0·27–0·35, silicon 0·10–0·35, manganese 0·5–0·7, nickel 2·3–2·8, chromium 0·5–0·8, molybdenum 0·4–0·7, sulphur 0·045 max., phosphorus 0·045 max., per cent.

Hardening and tempering treatment, and mechanical properties are specified: the latter include ultimate tensile stress 100 tons per sq. in. minimum.

HEAT- AND CORROSION-RESISTING MATERIALS

Scaling of Metals and Alloys in Sulphur-containing Gases

O. KUBASCHEWSKI and O. VON GOLDBECK: 'Scaling of Metals in Presence of Sulphur and Sulphur-containing Gases.' *Metalloberfläche*, 1954, vol. 8, Mar., pp. A33–A36.

Further to a discussion of the mechanism of scaling of alloys in air, and of its practical significance (*ibid.*, 1953, vol. 7, pp. A113–A118), the authors consider the problem of scaling in the presence of sulphur and sulphur-containing gases, which is a more complex phenomenon. The opening section of the paper comprises a review of some of the major factors involved in sulphur attack, demonstrating the difficulty which must be experienced in evolving materials which will withstand such conditions. The mechanism of sulphide formation is then discussed, on the basis of observations made by the authors and others on sulphidation in a variety of metals and alloys. It is pointed out that physical data available on the metal sulphides and sulphates are so meagre that any attempt to develop sulphur-resistant materials on a theoretical basis is unlikely to succeed. Progress has therefore been along the lines of application of the results of empirical tests, some typical examples of which are discussed. On the basis of the literature and their own experiments, the authors have compiled a table showing the order of resistance of metals and alloys to hydrogen sulphide, sulphur and sulphur dioxide. The paper also includes a table summarizing the properties of the sulphides of aluminium, magnesium, chromium, molybdenum, manganese, iron, nickel, cobalt, copper, silver, zinc, and lead.

Oxidation Characteristics of High-Purity Nickel

See abstract on p. 81.

Corrosion Tests in Regenerative Air Preheaters

J. F. BARKLEY, H. KARLSSON, A. A. BERK, C. F. STARK and L. R. BURDICK: 'Corrosion and Deposits in Regenerative Air Preheaters.' *U.S. Bur. Mines, Rept. of Investigations* 4996, Aug., 1953; 23 pp.

Two major problems in the operation of air preheaters for boilers are corrosion and the accumulation of deposits on the heating surfaces, necessitating, in the first case, all too frequent replacement of parts, and causing, in the second case, decrease in size of gas and air passages (with resultant pressure drop for a given gas flow), and adversely affecting heat transfer. These factors, singly or in combination, severely affect efficiency.

The field and laboratory investigations reported in this paper cover research on (1) the relative resistance to deterioration of various metallic materials, metallic coatings, and fired clays, and (2) examination of the nature of the deposits formed and their effects. The work is a co-operative effort of The Air Preheater

Corporation of New York, and the U.S. Bureau of Mines.

The materials exposed for test included (*a*) various grades of aluminium-base alloy, bronzes, low- and high-alloy steels (including a wide selection of austenitic nickel-chromium steels), copper, Hastelloy (nickel-molybdenum-base alloys), Inconel (nickel-chromium alloy containing a small percentage of iron), Monel (nickel-copper alloy) Stellite (cobalt-chromium-base alloys), titanium, and (*b*) various types of coating, including copper+nickel+chromium, nickel+chromium, nickel-zinc alloy and zinc plating, and a number of non-metallic finishes. Cor-Ten low-alloy steel (containing small percentages of chromium, copper and nickel) was used as the standard of comparison.

Field tests were made under regular operating conditions, in an air preheater serving an underfeed stoker-fired water-tube boiler. The plant equipment is described, and pertinent operating data for the 5½-year period of the tests are tabulated. Major, minor, and trace constituents, percentages of sulphates, solubilities in water, acidities, physical formations and colour of typical deposits are listed, and rates of corrosion of the individual materials are tabulated. Results are also given of laboratory experiments made to determine resistance to attack by sulphuric acid under various conditions. Structure and grain-size of some of the materials are correlated with other data.

The report closes with some recommendations for prevention of corrosion and accumulation of deposits. In this connexion it is suggested that the following remedial measures would be helpful:—reduction of sulphur in the fuel used; reduction of moisture in the air and in the fuel: use of additives such as $Ca(OH)_2$, $CaCO_3$ and $MgCO_3$, to the fuel, the flue gases, or the air; removal of dust from the flue gases before they enter the air preheater, removal of deposits during operation of the plant.

Heat-Resisting Materials of Medium Alloy Content

R. W. BORING: 'Low-Cost Alloys Offer Good Heat-Resistance.' *Iron Age*, 1954, vol. 173, Mar. 11, pp. 146–8.

The writer points out that although for heat-resisting applications of extreme severity high-alloy materials are essential, less drastic conditions of service can in many cases be adequately met by use of materials of somewhat lower alloy content. From this point of view he then summarizes properties typical of the following grades of steel:

A.I.S.I. 309: carbon 0·20 max., manganese 2·00 max., silicon 1·00 max., chromium 22·0–24·0, nickel 12·0–15·0, per cent.

A.I.S.I. 310: carbon 0·25 max., manganese 2·00 max., silicon 1·50 max., chromium 24·0–26·0, nickel 19·0–22·0, per cent.

A.I.S.I. 430: carbon 0·12 max., manganese 1·00 max., silicon 1·00 max., chromium 14·0–18·0, per cent.

A.I.S.I. 446: carbon 0·35 max., manganese 1·00 max., silicon 1·00 max., chromium 23·0–27·0, per cent.

Tables show physical and mechanical properties of these steels, including both short-time and creep characteristics at elevated temperatures. The properties are discussed in relation to suitable high-temperature applications, and some comparison is made with R.A. 330, which was the subject of part I of the article (*ibid.*, Feb. 18, pp. 137–41; *Nickel Bulletin*, 1954, vol. 27, No. 4, p. 70).

Metallurgy for Engineers: Textbook

See abstract on p. 93.

Diffusion of Chromium and Other Elements into Nickel and Nickel Alloys

See abstract on p. 83.

Porcelain and Ceramic Coatings on Metallic Materials

See abstract on p. 80.

Shell Moulding of Stainless Steel

H. J. COOPER and M. L. KATZ: 'Casting Stainless Steel in Shell Molds.' *Metal Progress*, 1954, vol. 65, Apr., pp. 102–6: cf. *Precision Metal Molding*, 1953, vol. 11, Oct., pp. 44–5.

This article is based on the practice of Cooper Alloy Foundry Company, Hillside, New Jersey, U.S.A., who have been in the forefront of development of the shell-moulding process, specializing in the production of high-alloy castings. This process utilizes the thermo-setting properties of phenolic or urea resins to provide a bond for silica or zircon sand, in the construction of a mould. While the exact mixtures vary from one plant to another, experience in the Cooper foundry shows that in shell-moulding of high-alloy materials a mixture containing sand 87, resin 8, iron oxide or zircon flour 5, per cent., is the most economical for obtaining consistent results. The sand used is of 160 A.F.S. fineness.

The mechanics of the shell-moulding process are described in this article by means of a step-by-step series of seven photographs: this description is followed by a discussion of the 'know-how' or 'art' of the process, and of metallurgical considerations involved in shell moulding. Advantages of the process are enumerated, the degree of dimensional accuracy obtainable is emphasized, and prospects of future development are briefly discussed.

Effect of Heat-Treatment on Magnetic and Other Properties of Cold-Worked Austenitic Steel Wire

S. STORCHHEIM: 'Effect of Heat-Treatment on the Magnetic Properties of Cold-Worked 18-8.' *Wire and Wire Products*, 1954, vol. 29, Feb., pp. 147–50.

The information presented is similar to that given in *Iron Age*, 1953, vol. 172, Oct. 1, pp. 112–14: see abstract in *Nickel Bulletin*, 1953, vol. 26, No. 12, p. 215.

Nickel Alloys in Needle Thermocouples

See abstract on p. 80.

Stainless Steel in Bone Surgery

F. P. BOWDEN, J. B. P. WILLIAMSON and P. G. LAING: 'Metallic Transfer in Screwing and its Significance in Bone Surgery.' *Nature*, 1954, vol. 173, Mar. 20, pp. 520–2.

Experiments *in vitro* and *in vivo* have established that the most suitable material for metallic plates and other repair parts used in bone surgery is a stainless steel of the following composition: carbon 0·07, silicon 0·30, manganese 0·5, chromium 18·0, nickel 8·0, molybdenum 2·75, per cent., iron balance.

Screwdrivers, spanners and forceps used in bone surgery are also made from stainless steel, but of the straight 13½ per cent. chromium type, and it is shown in this paper that metallic transfer from the high-chromium to the austenitic nickel-chromium steel, occurring during insertion of the plates, screws, or bolts, may result in the presence of free iron in the tissues adjacent to the parts which have been in contact with the chromium-steel tools. The presence of the free iron is evidence of corrosion of the stainless steel, and such a condition has been found to be accompanied by an 'electrolytic inflammation' which interferes with bone union and healing. The inert material can thus, on occasion, behave as a toxic one. Physical experiments made by the authors show that it is almost impossible to insert a screw without leaving some fragments of metal from the screwdriver adhering to the screw, and the amount so transferred increases markedly if slip occurs during screwing. Although the steel used for the plates, screws, etc., is highly resistant to corrosion by body fluids and tissues, the straight-chromium steel of the tools is not equally inert, and an appreciable potential can be set up between these two steels, with the result that they form an electrolytic couple which may act as a centre of corrosion, and have a harmful influence on healing. The effect of differential transfer can, it is found, be lessened by use of Vitallium-type alloy for the driver and the screws.

Huey Boiling Nitric-Acid Test: Influence of Contamination of Solution

R. J. BENDURE: 'Contamination of Nitric Acid as a Source of Error in the Huey Boiling Nitric-Acid Test.' *Corrosion*, 1954, vol. 10, Mar., N.A.C.E. News, p. 1.

In the standard boiling nitric-acid test the rate of attack, on most materials, follows a regular pattern. In the first period the rate is sometimes slightly higher than in the second one, and in each of the three succeeding periods the rate of attack is as high or higher than in the one immediately preceding it.

Following some irregularities in penetration rates observed in the laboratories of Armco Steel Corporation, an examination was made to determine possible sources of error. It was ultimately discovered that, from time to time, solutions of hydrofluoric and sulphuric acids were being evaporated to dryness under one of the hot plates on which the Huey tests were being made, and to confirm that this might be the disturbing factor the following experiments were carried out:—Huey tests were made while a mixture of hydrofluoric and sulphuric acids was evaporated under the hot plate, and simultaneously a high humidity was maintained within the hood, by boiling a large container of water. Abnormally high rates of attack were observed on all the specimens exposed in such conditions, whereas subsequent periods of test in the absence of sulphuric and hydrofluoric acids yielded normal results. Qualitative examination of the nitric-acid solution which had caused high rates of attack showed the presence of an appreciable quantity of fluorides.

The author states that the influence of contamination of the nitric acid used for the Huey test is confirmed by a report from a plant laboratory in which erratic rates of attack were eliminated by discontinuance of the use of hydrogen sulphide in the vicinity of the test flasks.

Corrosion-Resistance and Other Properties of Nickel-Molybdenum-base Alloys

W. A. LUCE: 'Chlorimet 2.' *Chemical Engineering*, 1954, vol. 61, Feb., pp. 246–52. (Even numbers only.)

This alloy, described in the Corrosion Forum series, contains nickel 62, molybdenum 32, iron 3 max., silicon 1, carbon 0·10, per cent.: it is closely similar to some grades of the Hastelloy, Corrosist and Langalloy alloys. Such materials give specially good resistance to corrosion by acids, particularly hydrochloric acid. Due to the fact that they contain no chromium, they are suitable for use in reducing or neutral media only, *not* in oxidizing conditions. Resistance to corrosion is illustrated by a series of charts similar to those employed in assessing the properties of the other materials which are being described in this series of articles. Physical and mechanical properties are tabulated and some applications are discussed.

See also—

W. A. LUCE: 'Chlorimet 3.' *ibid.*, Mar., pp. 254–60 (even numbers only).

Chlorimet 3 (also similar in type to certain grades of the Hastelloy, Corrosist and Langalloy alloys), has the following nominal composition: nickel 60, chromium 18, molybdenum 18, iron 3 max., silicon 1, carbon 0·07 max., per cent. Like Chlorimet 2, the alloy provides good resistance to attack by many special media for the handling of which other alloys have proved inadequate. Chlorimet 3 retains a good degree of resistance to reducing corrosives, and in addition, by virtue of its chromium content, is satisfactorily resistant to many severely oxidizing corrodents. Its characteristics in relation to the various corrosives are charted, and its physical and mechanical properties, and typical applications, are described.

Heat-Treatment of Welds in Austenitic Steels

'Stress-Relieving Austenitic Weldments.' *Steel*, 1954, vol. 134, Mar. 1, p. 100.

This short note is based on recommendations which have recently been issued by the American Welding Society.

It is pointed out that the problem of stress-relief

treatment involves three important variables: (1) structural characteristics of the weld, (2) service requirements, and (3) composition of the austenitic material welded. The recommendations quoted below may be broadly considered as applicable to steels of the A.I.S.I. 300 series. The inherent rigidity of a weldment must be a major factor in deciding whether the joint should be heat-treated and, if so, the nature of the treatment. Many structures, for example thin-walled vessels, will not require treatment, due to the relative ease of deflection and the consequently low level of residual stress: conversely, thick-walled vessels, where a high degree of restraint is present, will contain higher levels of residual stress and will be more likely to require stress-relief. Symmetry of the welds, the combination of thin and thick sections, the type of weld made, and the cyclic or steady nature of the loading to be borne are further factors which must influence a decision on treatment.

Subject to the foregoing considerations, the following recommendations are made:—

'*When to Treat—*

No heat-treatment required:—

(1) Where corrosion-resistance, as welded, is adequate.
(2) Where high-temperature strength is the primary service requirement, and in the absence of serious stress concentrations.
(3) In relatively thin weldments.

Heating of the weldment or structure to a minimum of 1750°F. (955°C.) is recommended:—

(1) Where dimensional stability must be assured.
(2) To improve the corrosion-resistance of the weldment.
(3) Where improved mechanical properties are desired in high-temperature service.

How to Treat—

Rate of heating of the weldment during the postweld heat-treatment, except for very thin members, should not exceed 300°F. (166°C.) per hour to 1100°F. (598°C.) above which temperature the rate should not exceed 600°F. (332°C.) per hour. Holding time at temperature should be 1 hour per inch of thickness. For thicknesses under 2 inches, 2 hours is minimum holding time. Cooling to 1000°F. (538°C.) from the heat-treating temperature should be as rapid as practicable for the unstabilized grades.

Moderate air-cooling should be used for the stabilized grades. Cooling from 1000°F. (538°C.) may be in still air. However, in the case of heavy-walled weldments or structures of non-uniform cross-section, the cooling rate should not exceed 400°F. (222°C.) per hour divided by the wall-thickness in inches.

Local heating should be done only for improvement of mechanical properties. Such local heat-treatments will result in reduced corrosion-resistance in the temperature transition zones for any heat-treatment above 850°F. (455°C.).'

Chromium-Nickel Steel Electrodes for Welding High-Chromium Steels

J. A. McWILLIAM: 'The Welding of Certain Heat-Resisting Steels.' *Trans. Inst. Welding*, 1953, vol. 16, Aug., pp. 96–9, 110.

The author reports tests made to compare the characteristics, in metal arc welding, of a low-carbon 13 per cent. chromium steel and of a similar steel containing 0·16 per cent. of aluminium. Pre-heating and post-heating treatments were carried out by methods normally used in welding shops. The electrodes used were: (1) 14 per cent. chromium steel, (2) 18–8–Nb chromium-nickel steel, and (3) 25–17 chromium-nickel steel. The best results were obtained with the 25–17 per cent. electrode: tests made on welds prepared with such electrodes are fully recorded. No cracks were detected in the welds: the parent metal was not hard, and the tensile properties were satisfactory. The author also refers to a case of failure in a weld in a 25–20 chromium-nickel steel fan impeller, attributed to bad penetration.

Production and Processing of Clad Metals

See abstract on p. 80.

Gas-Cutting of Stainless Steel

J. R. KIRWIN and J. HOLMSTOCK: 'Uniform Powder Flow Gives Better Cuts in Stainless.' *Iron Age*, 1954, vol. 173, Mar. 4, pp. 156–7.

This short article describes improved equipment for cutting of stainless steel. Its chief characteristic is the employment of a closely controlled rate of powder flow, which is secured by means of a vibratory hopper, working independently of air flow. From this hopper, powder is metered accurately by an electrical vibrator into a stream of compressed air, which transports it to the cutting torch through a separate rubber hose. Control over the flow of powder is obtained by increasing or decreasing the resistance in the electrical circuit, by means of a variable transformer. The flow of powder from the vibratory feeder is not readily affected by the density or particle size of the powder, and the powder-dispensing equipment can also tolerate slight amounts of moisture in the powder without harmful effects on powder flow.

Cadmium Plating of Stainless Steel

See abstract on p. 92.

Attention is drawn to the fact that many of the names of materials mentioned in The Nickel Bulletin are *Registered Trade Marks.*

THE MOND NICKEL COMPANY LIMITED
SUNDERLAND HOUSE · CURZON STREET · LONDON, W.1

PRINTED IN ENGLAND *by* WIGHTMAN MOUNTAIN LTD.
WESTMINSTER, LONDON, S.W.I